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Review

Current and future applications for nanofiltration technology in the food processing

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ABSTRACT

The increase in energy costs and the demands for products with greater nutritional value and of processing procedures less toxic to the environment are attractive factors for transferring membrane processing to food industries. Nanofiltration (NF) technology is still evolving, finding more and more applications in food processing and appears as an important alternative to conventional methods. The purpose of this review is to present the recent development and future potential of NF processes in the food industry. Recent research has highlighted the potential for NF use in wide ranging, including water softening, wastewater treatment, vegetable oil processing, beverage, dairy and sugar industry. NF has been established as greater separation efficiency, successfully reduces the wastewater, done under low temperatures, reduction in number of processing steps and presents a promising choice toward the achievement of cost effective process. NF carries quite distinctive properties such as pore radius and surface charge density which influences the separation of various solutes.

Keywords: Beverage; Dairy; Drinking water; Sugar industry; Vegetable oil; Wastewater treatment

1. Introduction

Membrane separation processes are used to concentrate or fractionate a liquid to yield two liquids that differ in their composition. It stands out as alternatives to conventional processes for the chemical, pharmaceutical, biotechnological and food industries (Cassano et al., 2003; Baker, 2004; Jiao et al., 2004; Ravanchi et al., 2009; Aroon et al., 2010; Lau et al., 2012). In many cases the low energy consumption, reduction in number of processing steps, greater separation efficiency and improved final product quality are the main attractions of these processes (Cheryan, 1998; Baker, 2004; Aroon et al., 2010; Cuartas-Uribe et al., 2010; Lau et al., 2012). NF can also be applied for more challenging applications, involving fractionation rather than purification. The nature of the membrane controls which components will permeate and which will be retained, since they are selectively separated according to their molar masses or particle size (Cheryan, 1998). It is well known that NF membranes can be used for salt fractionation since the rejection of monovalent salts is lower than that of multivalent salts (Tanninen et al., 2006; Hilal et al., 2007). An extreme case of charge-induced separation is the observation of negative rejections of monovalent ions in the presence of multivalent ions or polyelectrolytes (Van der Bruggen et al., 2003; Ting et al., 2007). NF appears as an important alternative to conventional methods of food processing.

This review summarizes the recent developments and future potential of NF membrane processes in the food industry that appear to have great potential in the production of high quality food, including water softening, wastewater treatment, vegetable oil processing, beverage industry, dairy industry and sugar industry.

2. Membrane

The separation performance of a membrane is influenced by its chemical composition, temperature, pressure, feed flow and interactions between components in the feed flow and the

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membrane surface. The four major pressure driven membrane processes are ultrafiltration, reverse osmosis, microfiltration and nanofiltration; brief general descriptions of the membrane processes used in the food industry are provided in this section.

2.1. *Pressure driven membrane processes*

A hydrostatic pressure gradient is the driving force used to achieve the desired hydrodynamic flow through the membrane (and through a deposited layer that may develop during the filtration process). In some cases, concentration gradients or electrical potential gradients may also be used as additional driving forces (Cheryan, 1998; Baker, 2004). Four pressures driven membrane processes are distinguished in practice:

2.1.1. *Microfiltration (MF)*

MF is a membrane process that involves the use of membranes with a pore size of 0.2–2 μm, and can selectively separate particles with molecular weights of >200 kDa. MF uses pressures lower than 0.2 MPa and separates molecules between 0.025 and 10 μm. MF is primarily used to separate particles and bacteria from other smaller solutes (Baker, 2004; Hua et al., 2007).

2.1.2. *Ultrafiltration (UF)*

UF involves the use of membranes with a molecular weight cut off (MWCO) in the range of 1–300 kDa and a pore size of ~0.01 μm. UF uses pressures greater than 1 MPa and used to separate colloids like proteins from small molecules like sugars and salts (Baker, 2004).

2.1.3. *Nanotiltration (NF)*

NF lies between the separation characteristics of reverse osmosis (RO) and UF process which is widely used for several applications such as water softening and wastewater treatment. The pore size of the NF is in the range of 0.5–1 nm. It concentrates, fractionates or purifies aqueous solutions of organic solutes with molecular weight between 100 and 1000 Da and mixture of monovalent/multivalent salts and uses pressures between 1 and 4 MPa (Baker, 2004; Salehi et al., 2011). Since, the NF membrane carries negative charge at the surface, positive charged ions will be attracted and negative charge will be repelled due to Donnan effect. The most successful NF models are those based on the combination of the extended Nernst–Planck equation with the Donnan steric equilibrium. These models have been typically solved by using iterative procedures based on the Runge–Kutta method (Vezzani and Bandini, 2002; Kumar et al., 2013). Most NF membranes are composite materials supported by polymer substrate and manufactured in a spiral wound design as opposed to a flat sheet or tube geometry. The predominant model used today for industrial applications is the spiral configuration. Polyamide (PA) is used as the thin film membrane layer in NF membranes (Baker, 2004; Hong et al., 2006).

2.1.4. *Reverse osmosis (RO) or hyperfiltration*

RO membranes are characterized by a MWCO of ~100 Da, and the process involves pressures 5–10 times higher than those used in UF. It uses pressures between 4 and 10 MPa and concentrates particles with molar masses below 350 Da and this technique reject nearly all solutes and desalinate water (Baker, 2004).

2.2. *Operational parameters*

The main physical operational parameters that affect the permeate flow rate are: pressure, temperature, viscosity and density of the feed fluid, and the tangential velocity (Scott, 2003).

The viscosity can be controlled by two factors: solids concentration in the feed and temperature (Hwang and Kammermeyer, 1998). An increase in feed concentration alters the viscosity, density and diffusivity of the feed solution, causing a decrease in permeate flow rate (Satyanarayana et al., 2000). An increase in temperature results in a decrease in fluid viscosity and increase in molecular mobility, that is, in diffusivity. For its part, an increase in tangential velocity increases the permeate flow rate by provoking greater turbulence, causing a dispersion in the solute molecules concentrated on the membrane surface, reducing the thickness of the gel layer (Cheryan, 1998; Cheng and Lin, 2004). There is a linear relationship between flow rate and the inverse of the solvent viscosity for NF and UF membranes, indicating that the main mass transport mechanism in these systems is convection (Tsui and Cheryan, 2004).

3. Water softening

In the past few years, increasing water scarcity and deteriorating water quality are becoming growing problems in many regions of the world (Loo et al., 2012). NF and RO are used for a wide range of applications, such as the purification of water to produce potable water (mainly sea and brackish water desalination), rejection of pesticides and the production of ultrapure water for the semiconductor industry (Ghaemi et al., 2012). During the last decade, the interest in the use of membrane processes in general and NF in particular has emerged in wastewater treatment as well as drinking water and process clean water production (Table 1). This growth can be explained by a combination of (1) growing demand for water with high quality, (2) growing pressure to reuse wastewater, (3) better reliability and integrity of the membranes, (4) lower prices of membranes due to enhanced use, and (5) more stringent standards, e.g. in the drinking water industry (Van der Bruggen et al., 2008; Greenlee et al., 2009).

Further improvements can be achieved through the introduction of NF as RO pretreatment process. Since NF retains turbidity, microorganisms, hardness, the most part of multivalent ions, and 10–50% of monovalent species, as a consequence, the osmotic pressure of the RO feed is decreased thus allowing the unit to operate at higher recovery factors. Coupling RO and NF for seawater desalination, a global recovery factor of 52% can be obtained (higher than that of a typical RO operation which is in the range of 35–40%) (Macedonio et al., 2007). Moreover, the integrated NF–RO process is more environmentally friendly, because fewer additives (antiscalants and acid) are needed (Van der Bruggen and Vandecasteele, 2002).

Oh et al. (2000) developed a membrane process that uses an NF module coupled to a stationary bicycle to generate energy required for pressurizing the feed. However, fouling of the membranes results in a reduction in water flux, which leads to higher treatment costs. Membrane fouling also limits the water recovery, i.e. the ratio of permeate to feed stream, to values of about 80% in the drinking water industry (Nederlof et al., 2005; Amoudi, 2010; Sutzkover-Gutman et al., 2010). Nilson and DiGiano (1996) reported that the hydrophobic fraction of natural organic matter in surface water caused almost all

Table 1 – Pre-treatment and post-treatment of water using NF and RO process.

Production step	Membrane processes	Comments
Desalination/softening of process, boiler and cooling	NF/RO	RO removes minerals, particles plus most of the bacteria and pyrogens
Preparation of diafiltration water	RO	Diafiltration water is high-quality water in accordance with process water standards
Pyrogen removal	NF/RO	Membranes with MWCO less than 10,000 remove most pyrogen
Concentration of sugar water	RO	Concentration of sugars to reduce BOD. Water and sugars might be recycled in the process
Condensate polisher	NF/RO	Concentration of the evaporator condensate, for example in case of carry-over with high BOD/COD
Concentration of UF permeate	RO	UF permeate contains the low molecular components such as sugars and salts

Source: adapted from Lipnizki (2010).

fouling of a polysulfone (PS) NF membrane, while the hydrophilic fraction showed less fouling. It is also widely acknowledged that hydrophobic membranes have a higher tendency to foul than hydrophilic membranes.

Typically, the rejection of a divalent ion of the same charge as the membrane is above 95%, whereas the rejection of a monovalent ion of the same charge can be anywhere between 20 and 80% (Schaep et al., 2001). Thus, NF membranes allow ion fractionation, which is a significant advantage and one of the reasons of the fast commercial growth of the process (Greenlee et al., 2009).

Geluwe et al. (2011) investigated whether the decomposition of natural organic matter in the concentrate stream by O_3, has a positive effect on the membrane flux of four NF membranes (NF 90, NF 270, Desal 51, NF-PES 10). The results show that O_3 oxidation causes a significant alleviation of membrane fouling for all investigated membranes.

These NF membranes are recommended for use in water softening systems: UOP Fluid Systems modules 8231-LP (cellulose acetate blend) and 8921-UP (TFCS PA), Filmtec NF 70 and NF 40, Toray modules SCL-100 (modified cellulose acetate) and SU 600 (composite PA), Nitto Denko NTR-729 HF, Desalination Systems Desal-5 and DuPont SM15 (Nunes and Peinemann, 2001; Van der Bruggen et al., 2008). The characteristics of commercially available NF membranes are summarized in Table 2.

4. Wastewater treatment

The food industry is one of the largest water-using industries. The water used in the food industry can be generally classified into three types: process water, boiler and cooling water, general purpose water (water to rinse raw materials, prepared products, and equipment). In Table 1, some applications of membranes in the pretreatment and post-treatment of water are summarized. The potential for NF in wastewater treatment and water reuse is noteworthy (Frenzel et al., 2006; Manttari et al., 2006; Bellona and Drewes, 2007; Shirazi et al., 2010), but hindered by unstabilities in operation caused by membrane fouling. Extensive research projects in which NF was used for water reclamation have been carried out; in the majority of these, membrane fouling was studied as a potential problem. Industrial plants may be successful (Cassano et al., 2001; Frank et al., 2002), but their success depends on a thorough understanding of possible interactions between the feed solution and the membrane, causing organic fouling, biofouling, scaling, or particulate fouling. When wastewater is to be treated, the concentrate is usually another problem (Van der Bruggen et al., 2003; Samhaber, 2005).

Membrane processes have been claimed to have a good potential of development to recover valuable compounds in seafood processing industries (Massé et al., 2008; Bourseau et al., 2009; Walha et al., 2009). Cooking waters from buckies, shrimps and tuna have a high level of polluting load (chemical oxygen demand (COD) between 5 and 40 g O_2/l) and have to be treated before being rejected in the environment. However, these juices seem to contain interesting flavor compounds. This recovery would allow the industrialists to diminish the waste water treatment cost and to recover high added value molecules (Lin and Chiang, 1993; Vandanjon et al., 2002). Vandanjon et al. (2002) used a membrane process system to reduce the pollution load and to concentrate flavor compounds of seafood cooking juices (buckies, shrimps and tuna). NF did not seem to be efficient enough for flavor recovery, whereas RO was, for shrimps and buckies cooking juices. As for tuna or shrimps, COD reduction of 80% by NF and 95% by RO seems to have promising results. It would be now interesting to experiment a very selective RO membrane or to combine NF and RO for a complete elimination of COD (Cros et al., 2006). Tuna cooking juices contain high organic load preventing the rejection in the environment without treatment. But the effluents present an interesting fishy odor and it is worth recovering aroma compounds. NF sharply decreases the global intensity of juices and modifies their aromatic equilibrium. However, the main characteristics and the marine nature of juices were kept. In Walha et al. (2011) work, aromas were concentrated from highly salted tuna cooking juices by NF (AFC30, PCI membrane). A pre-treatment by MF induces a marked increase in permeation fluxes during NF concentration while it slightly affects the aromatic properties of juice. The NF membrane characteristics, as provided by the manufacturer and the retention of different ions are shown in Table 3 (Nyström et al., 1999; Balannec et al., 2005).

5. Beverage industries

One of the promising applications of NF in the food industry is related to beverage industries. Small monovalent ions and water will to some extent pass a NF membrane, while larger molecules such as sugars are retained. Fruit juices have been traditionally concentrated by multi stage vacuum evaporation, resulting in a loss of fresh juice aroma, changes in color and a cooked taste due to the thermal effects. Heat treatment of a juice influences both the color and odor when evaluating the juice by sensory analysis (Nabetani, 1996; Jiao et al., 2004; Madaeni and Zereshki, 2010). Technological advances related to the development of new membranes and improvements

Table 2 – The characteristics of commonly used NF membranes (data provided by the manufacturers).

Membrane type	Material	Configuration	Range of pH tolerance	Maximum temperature	Maximum pressure	Retention (%)/or maximum molecular mass retained	Water permeability/or maximum Feed Flow
AFC99 (PCI Membranes Ltd., UK)	PA film	Tubular	1.5–12	80 °C	6.4 MPa	99% NaCl	–
AFC80 (PCI Membranes Ltd., UK)	PA film	Tubular	1.5–10.5	70 °C	6 MPa	80% NaCl	–
AFC40 (PCI Membranes Ltd., UK)	PA film	Tubular	1.5–9.5	60 °C	6 MPa	60% $CaCl_2$	–
AFC30 (PCI Membranes Ltd., UK)	PA film	Spiral wound	1.5–9.5	60 °C	6 MPa	75% $CaCl_2$	
NF-90 (FilmTec™ Membranes, USA)	PA film	Spiral wound	2–11	45 °C	4.1 MPa	92% NaCl	8.3 l/h m^2 bar
NF-270 (FilmTec™ Membranes, USA)	PA film	Spiral wound	3–10	45 °C	4.1 MPa	70.6% NaCl	13.8 l/h m^2 bar
NF-200-400 (FilmTec™ Membranes, USA)	PA film	Spiral wound	3–10	45 °C	4.1 MPa	35–50% $CaCl_2$	15.9 m^3/h
NF-2540 (FilmTec™ Membranes, USA)	Polypiperazine amide film	Spiral wound	3–10	45 °C	4.1 MPa	98% $MgSO_4$	1.4 m^3/h
ESNA1-LF2 (Nitto Denko, Osaka, Japan)	Composite PA	Spiral wound	3–10	45 °C	4.16 MPa	73–92% $CaCl_2$	17 m^3/h
ESNA1-LF (Nitto Denko, Osaka, Japan)	Composite PA	Spiral wound	3–10	45 °C	4.16 MPa	84–96% $CaCl_2$	17 m^3/h
ESNA1-LF-LD (Nitto Denko, Osaka, Japan)	Composite PA	Spiral wound	3–10	45 °C	4.16 MPa	86–95% $CaCl_2$	17 m^3/h
ESNA1-LF2-LD (Nitto Denko, Osaka, Japan)	Composite PA	Spiral wound	3–10	45 °C	4.16 MPa	83–90% $CaCl_2$	17 m^3/h
MPS-34 (Koch Membrane Systems, USA)	Polysulfone composite	Spiral wound	0–14	70 °C	3.5 MPa	200 Da	–
MPF-44 (Koch Membrane Systems, USA)	Polydimethylsiloxane	Spiral wound	–	40 °C	–	98% (5% lactose)	1.3 l/h m^2 bar
MPF-55 (Koch Membrane Systems, USA)	Polydimethylsiloxane	Spiral wound	–	40 °C	–	700 Da	1.0 l/h m^2 bar
N30F (Nadir Filtration, Germany)	PES	–	0–14	95 °C	–	26.7% NaCl	3.9 l/h m^2 bar
NF-PES-10 (Nadir Filtration, Germany)	PES	–	0–14	95 °C	–	15.2% NaCl	12.9 l/h m^2 bar
Desal-5-DK (Osmonics, USA)	PA	–	–	50 °C	–	98% $MgSO_4$	5.4 l/h m^2 bar
Desal-5-DL (Osmonics, USA)	PA	–	–	50 °C	–	96% $MgSO_4$	9 l/h m^2 bar
StarMem-120 (Membrane Extraction Technology, UK)	Polyimide	–	–	60 °C	–	–	1 l/h m^2 bar
SS-01 (SolSep BV, Netherlands)	–	–	–	150 °C	–	97% (MW ~ 1000 in acetone)	10 l/h m^2 bar

Table 3 – Characteristics of some NF membranes in comparable conditions.

Membrane	Permeability (m^3/m^2 day MPa)	Cut off (g/mol)	Retention (%)										
			Fe^{3+}	Cr^{3+}	Ni^{2+}	So_4^{2-}	NO^{3-}	F^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	Citrate
Desal-5 DL (Osmonics, USA)	1.3	150–300	89.8	90.1	90.4	64.8	36.8	61.3	69	68	99.9	99.4	>99.9
Desal-5 DK (Osmonics, USA)	2.2	–	–	–	–	–	–	–	68	62	99	97.4	–
PVD-1 (Nitto Denko, Osaka, Japan)	0.8	180	96.0	96.4	96.4	71.4	24.8	69.6	–	–	–	–	–
NF 45 (Filmtec™ Membranes, USA)	1.2	–	99.0	99.6	99.7	56.9	25.0	71.0	52	58	99.3	99.1	–

in process engineering have been proved to overcome this limitation. It shows great promise in mild low temperature treatment of fruit and plant juices and extracts for clarification, microbial stabilization and concentration with low energy consumption. NF has been used for concentration of various must and juices (Nabetani, 1996; Warczok et al., 2004; Sotoft et al., 2012). In addition, NF process can be used in the production of the fermented food products, for example beer and wine. Membranes have initially established themselves as a clarification step after the fermentation. It can be predicted that the use of NF processes will bring great changes in the beverage industry in the future, with the development of membrane science and technology.

5.1. *NF for juice concentration*

The state-of-the-art process for juice concentration includes severe and energy consuming unit operations such as evaporation at high temperatures (up to 90 °C) with several disadvantages: loss of aroma, nutrients (vitamins and antioxidants), coloring due to maillard reactions, induction of cooked odor due to furfural formation, foam formation and large amount of energy needed for the removal of water (Sotoft et al., 2012). The promising alternatives are RO and NF membrane concentration. However, it cannot reach concentrations larger than 25–30°Brix with a single-stage RO system due to high osmotic pressure limitation, which is quite below the value of 45–65°Brix for standard products obtained by evaporation (Jiao et al., 2004). On the other hand, replacing RO by NF can improve the efficiency of the process, first, because the high pressure required in RO can damage the juice and second because the cost of RO is higher.

NF membrane techniques make it possible to obtain a product that is very similar to fresh juice, and it reduces and simplifies the clarification process. Nabetani (1996) developed an integrated RO–NF membrane system for highly concentrated fruit juice. Using this system to concentrate fruit juice from 10°Brix to 45°Brix, energy savings of 1/8 and 1/5 by this system can be obtained when compared with evaporation and freeze concentration, respectively.

Warczok et al. (2004) investigated concentration of apple and pear juice by NF at low pressures. During the experiments two tubular membranes (AFC80, PCI Membranes and MPT-34, Koch) and two flat-sheet membranes (Desal-5DK, Osmonics and MPT-34, Koch) were examined. The results show that the Desal-5DK membrane achieves a very high permeate flux, sufficiently high retention and a higher concentration degree than the MPT-34 membrane.

Another study, a conceptual process design with the use of integrated membrane processes is prepared for blackcurrant juice concentrate production to replace traditional multiple step evaporators and aroma recovery by Sotoft et al. (2012). The annual production scale is 17,283 ton of 66°Brix out of single strength juice. The operation cost is 0.40 €/kg blackcurrant juice concentrate, which is lower than the price of a traditional operation by about 43%. Therefore, the economical potential of the process is very promising and could supersede conventional evaporators.

5.2. *Beer and wine*

Initially, dead end filters were used in the production of fermented food products followed by the first trials of cross flow filtration for the clarification of beer and wine. In the

last decade, membrane filtration has established itself for the clarification of wine, and beer and based on its now proven reliability in other production steps. The demand for low-alcohol and alcohol-free drinks has been constantly growing over the last decade. NF can be used to reduce the alcohol concentration 8–10 times, while maintaining the beer flavor.

Membrane filtration has been applied to wine for a long time. As an alternative to chaptalization or other treatments, membrane can be applied to increase sugar contents in the wine without addition of nongrape components at ambient temperature and to adjust and balance the composition of the must. In particular, RO concentrates carbohydrates but also many other natural components of must, including malic acid that may emphasize the lack of wine sensory balance (Delfini et al., 1991). Furthermore, RO has high energy consumption and causes severe membrane fouling. These drawbacks could be solved by using permeoselective NF membranes. The NF process uses a pressure gradient to transport the grape must through the membrane and separates the permeate (mostly water) and the concentrate (fraction of enriched solutes). The use of NF membranes for the concentration of grape must may represent a further application of this technology. A preliminary comparison between NF and RO membranes for grape juice concentration was reported by Ferrarini et al. (2001). The use of NF leads to enrichment in tannins and organoleptic components by water reduction between 5 and 20%. However, applying this method to must from grapes of stalled maturity due to cold weather was found to be less effective, since apart from sugar, acid and green tannins are also concentrated (Smith, 2002; Massot et al., 2008). Versari et al. (2003) tested two NF membranes with the aim of increasing the sugar content of grape must use for wine production. In particular, the NF membranes provided a high rejection for sugars (range 77–97%), whereas the malic acid was retained to a low extent (range 2–14%). A two-stage NF process of grape must remove a permeate volume of 14%, thus allowed concentration of the sugars, i.e. the potential alcohol, to ca. 16%.

Wine is one of the most consumed alcoholic drinks in the world and moderate consumption of alcoholic beverages, especially red wine, is related to the decrease of cardiovascular diseases. Red wine consumption has higher antioxidant and cardiovascular protection benefits than other common alcoholic beverages (Szmitko, 2005). Ethanol plays an important physico-chemical and sensorial role in wine and its content is regulated by law (GUCE, 1999). The alcohol removal from wines has a great importance in the beverages industry due to the increasing demand on the non-alcoholic drinks market and also due to the increase of ethanol content of wine. Membrane processes can be used for removing the ethanol from a regular wine. Catarino and Mendes (2011) used membrane for producing wine with low alcohol content. Several membranes of RO (CA995PE, Alfa Laval) and NF (NF99 HF, NF99, NF97, Alfa Laval and YMHLSP1905, Osmonics) were used for removing ethanol from a 12 vol.% red wine. YMHLSP1905, NF99 and NF99 HF NF membranes showed higher effectiveness in alcohol removal from wine, due to their good permeability to ethanol and high aroma compounds' rejection, resulting in dealcoholized wine samples with promising organoleptic properties. NF membrane NF97 showed low flux as well as high ethanol rejection, making this membrane also unsuitable for the dealcoholization. The results of this study indicate that NF is effective for dealcoholizing wine and preserving its original characteristics. Labanda et al. (2009) studied the removal of ethanol from a model white wine using membranes of RO and NF. Their analysis was directed to the permeation of aroma compounds during the concentration process and they found high rejections of these compounds.

The elimination of wine "bad taste" appears to be a promising research subject. Ugarte et al. (2005) explored the methods for 4-ethylphenol and 4-ethylguaiacol reduction in red wines by combining NF and adsorption. The permeate obtained by NF is treated by hydrophobic adsorbent resins (XAD-16HP) and recycled up to the level of desired concentration. They claim that the same process could also be used for herby taste elimination. In principal, it should be possible to isolate the "bad taste" by using NF membranes, but for this elimination it would be necessary to improve the post-treatment (by adsorption and fining).

The tartaric acid stabilization of wines is usually done by cold treatment. A study suggests the use of NF and MF for tartaric acid stabilization. At first, the wine is concentrated by a NF until the precipitation of the tartar. The crystals are then eliminated by MF and the two permeates are gathered (California Energy Commission, 2001).

In addition, aqueous extracts from pressed distilled grape pomace were processed using UF and NF (MWCO of 350) membranes to obtain fractions enriched in compounds with antioxidant activity by Díaz-Reinoso et al., 2009. All the tested membranes presented similar rejections of total phenolics and sugars, and were suitable for concentration purposes. The phenolic content in retentates was increased by factors of 3–6 respect to the feed. The ethyl acetate soluble fraction of retentates presented high radical scavenging capacities, in the range reported for commercial antioxidants. Upon cleaning, membranes recovered 92–100% of their initial permeability.

6. Dairy industry

Membrane processes have been employed in the dairy industry for many years and represent a field of increasing interest. It allows improvements in the quality of existing dairy products, the development of new products, and enhanced process efficiency and profitability. Current applications are aimed at enhancing the manifestation of desired functional properties of milk proteins, MF/UF processes in fractionating caseins and whey proteins (Maubois and Ollivier, 1992), enhancing the microbial quality of dairy fluids using MF system (Daufin et al., 2001; Barbano and Elwell, 2006; Madaeni et al., 2011), upgrading the quality of low-quality whey (Kelly et al., 1992; Rosenberg, 1995), milk standardization and milk protein concentrate using UF membrane (Maubois, 1989; Puhan, 1992), NF applications in whey processing (Kelly et al., 1992; Rosenberg, 1995), recovery of lactose (Räsänen et al., 2002; Magueijo et al., 2005; Suárez et al., 2006; Cuartas-Uribe et al., 2010), whey demineralization (Lipnizki, 2010) and lactic acid separation (Milcent and Carrere, 2001; González et al., 2008). Combinations of membrane processes with traditional milk processing practices are being used to enhance the quality attributes of various dairy products.

6.1. *NF applications in whey processing*

Whey processing represents one of the first fields of application for membrane technology in the dairy industry. The potential applications of membrane technology in whey

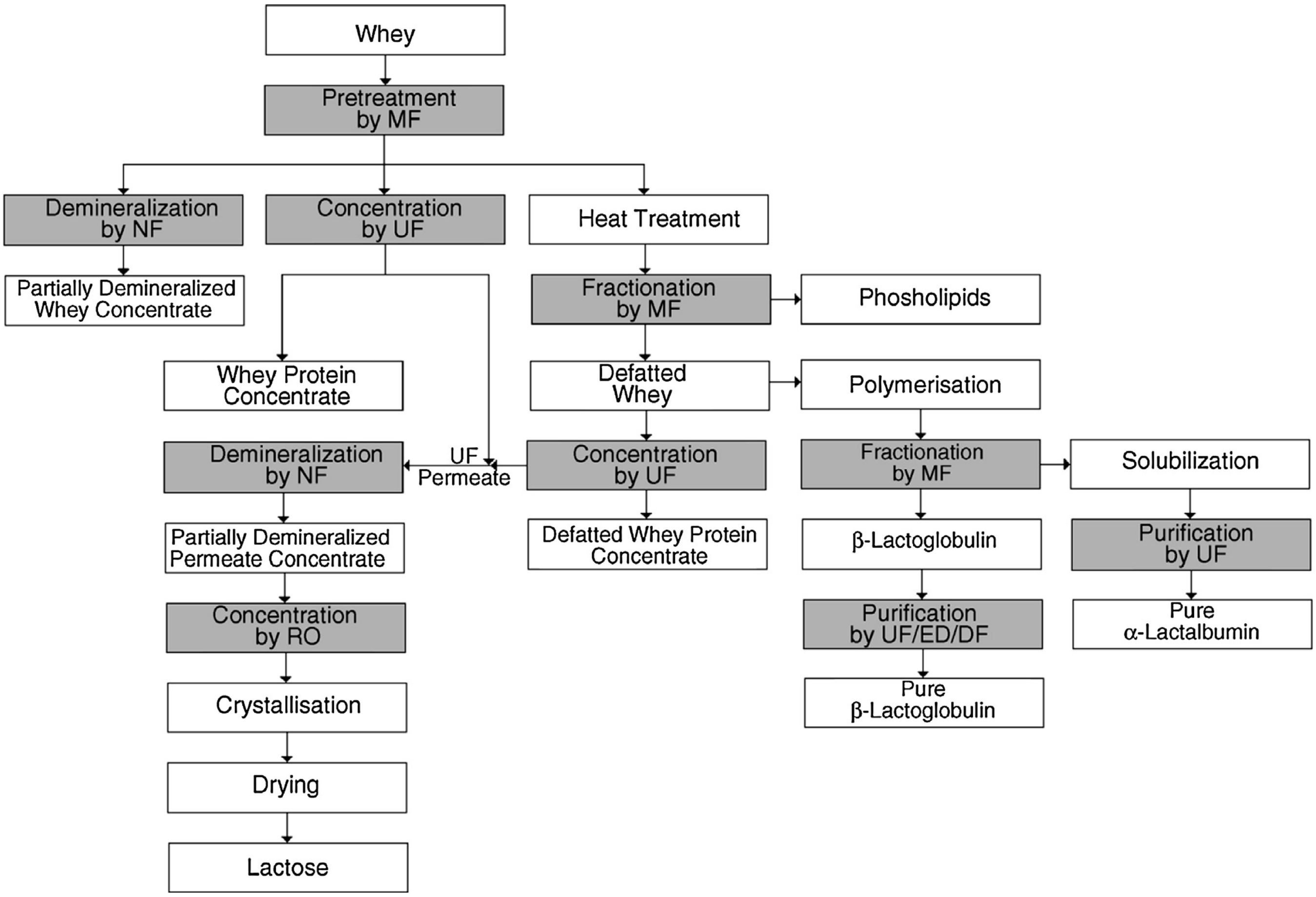

Fig. 1 – Applications of MF, UF, NF and RO in whey processing, as proposed by Lipnizki (2010).

processing are shown in Fig. 1. UF and RO have been extensively used in whey concentration and have allowed the development of a broad array of whey protein concentrates (WPC). Among the promising current applications for membrane technologies in whey processing are those aimed at increasing the protein content of WPC, fractionating whey proteins, and enhancing the manifestation of specific functional properties of whey proteins (Rosenberg, 1995).

In the dairy industry, the NF process is used to concentrate and partially demineralize liquid whey. Due to the selectivity of the membranes most of the monovalent ions, the organic acids, and some of the lactose will pass the membrane. NF is a very interesting alternative to ion exchange and electrodialysis if moderate demineralization is required (Lipnizki, 2010). Acid whey (pH 4.5–5) is produced in significant quantities during the manufacture of cottage cheese or quarg. Salt-containing whey is generated during the production of Cheddar cheese at stages following the application of salt to the curd. Acid and salty whey streams represent difficulties related to the functionality of the whey proteins they contain as well as to their adverse effects on the environment. NF offers simultaneous separation and concentration of minerals, and has been developed into applications such as the removal (84%) of salt from salty whey, the partial (42%) removal of acid from acid whey, and the partial demineralization of sweet whey in the manufacture of lactose or demineralized whey (Kelly et al., 1992). The maximum level of demineralization by NF is about 35% reduction of the ash content with a concentration factor of about 3.5–4. By applying a diafiltration step it is possible to increase the level of demineralization up to 45%. One advantage of NF compared to the other two processes is that NF is a simple process, concentration and partial demineralization of whey UF permeates prior to the manufacture of lactose and lactose derivatives and converting salt whey to normal whey while solving a disposal problem, treating cheese brine solutions to be reused (Lipnizki, 2010).

6.2. *Recovery of lactose*

Other applications of NF in whey processing involve the concentration and partial demineralization of whey UF permeate in recovery of lactose. Whey is a wastewater that shows a very high COD value (between 40,000 and 60,000 mg O_2/l), due to the presence of compounds such as lactose, proteins, vitamins, fat and mineral salts among others. Thus it cannot be drained without a treatment. Bioactive substances present in whey such as lactose and lactose derivatives have many applications in both the pharmaceutical and food processing industries (cheeses, drinks, soups, diet food and infant formulations) (Cuartas-Uribe et al., 2010).

Several authors worked in the utilization of NF to perform whey concentration and demineralization with different membrane operation process. Räsänen et al. (2002) and Suárez et al. (2006) worked in the concentration mode whereas Magueijo et al. (2005) worked in the total recirculation mode and concentration mode.

Magueijo et al. (2005) used NF membranes (NFT50 and HR-95-PP) for the recovery of cheese whey organic nutrients, resulting from “Serpa” cheese and curd production. The cheese whey is processed by NF to recover a rich lactose fraction in the concentrate and a process water with a high

salt content in the permeate. The concentration experiments showed that the selected membrane (NFT50) at 30 bar allows a water recovery of approximately 80%, concentrating the cheese whey nutrients approximately 5 times.

The aromatic PA spiral-wound NF membrane supplied by Osmonics (model DK2540C, USA) was used to carry out partial demineralization of whey and milk UF permeate by Suárez et al. (2006). Feed, permeate and retentate were analyzed for lactose, protein, ashes, ions (Ca^{2+}, Na^{+}, K^{+}, Mg^{2+}, P, Cl^{-}) and total dry extract content. By means of NF total salt content was reduced at least by 30%, depending on the operating conditions and volume concentration ratio.

Cuartas-Uribe et al. (2010) used the desal 5 DL membrane (150-300 MWCO, Dow Chemical, USA) to recover lactose from whey UF permeate. Lactose and ions rejection and permeate flux were measured using ultrafiltered sweet whey as feed solution. Three different operation modes (total recycle mode, concentration mode and continuous diafiltration mode) were used. Lactose retention was predicted by means of the Donnan Steric Partioning model (DSPM) and the Kedem–Spiegler model (KSM). The best fit for the total recycle and concentration modes was obtained with the KSM.

6.3. *Lactic acid separation*

Lactic acid (LA) is a versatile chemical used in food and chemical industries. LA is one of the major food preservatives and is also used for the manufacture of derivatives such as stearoyl-2-lactylate (dough conditioner). It can be manufactured either by chemical synthesis or by carbohydrate fermentation. The conventional process for fermentative production of LA is a batch process with low productivity and high capital and operating costs. LA fermentation is carried out at pH values between 4.0 and 6.0. The highest cost of the traditional process for LA production by lactose fermentation corresponds to the separation steps that are necessary to achieve the quality requirements for food grade LA (González et al., 2008).

Membrane processes such as NF and RO can be used in the cell separation step. Some authors have used NF or/and RO to remove LA from the fermentation broths, improving the fermentation yield using a membrane (Milcent and Carrere, 2001).

In case of an organic electrolyte the dissociation equilibrium is of great importance. The amount of undissociated LA and lactate anions present in the broth is based on LA equilibrium at the operating conditions. At pH values in the range of 5.5–6.0, which are typical values for industrial fermentations, LA is mainly found in the dissociated form, but if LA is the desired product pH around 3 is necessary. RO using thin-film composite membranes has been applied to concentrate dilute (1%) LA solutions. The LA rejection of the composite and cellulose acetate RO membranes used was found to be strongly dependent on pH (Schlicher and Cheryan, 1990).

Transport of LA through RO and NF membranes was studied by Timmer et al. (1993). A novel model, based on the extended Nernst–Planck equation, for the description of mass transfer of LA through these membranes was developed by authors. It can be used to predict mass transfer of LA under various pH and pressure conditions of the feed. The generalized model allows a simple calculation of the separation efficiency not only of LA but of other acids as well.

Another study, LA recovery from clarified fermentation broths by NF was studied by González et al. (2008). The electrostatic effect is a limiting factor in the recovery of LA by means of NF. LA transport through AFC80 (PCI Membrane) and DK2540C (Filtration Engineering) membranes was strongly affected by pH. Rejection increased with pH while flux decreased with this variable. However, at high feed concentrations, solute retention was much lower as the Donnan exclusion effect was attenuated. At the acidic pH required to convert lactate into undissociated LA (pH 3.0), the recovery of LA must be improved in order to reduce LA losses and increase purity and LA rejection was low (35–58%). Permeate flux increased with pressure and decreased with pH. Lactate rejection increased with pressure and pH. Lactate rejection of the DK2540C membrane at 60 l/h m^2 was 10–91% in the pH range of 2.7–6, whereas lactate rejection of the AFC80 membrane was 45–82%.

7. Sugar industry

Sugar processing is one of the most energy-intensive processes in the food industry. Therefore, membrane separation processes like MF and UF of raw sugar juice (Hinkova et al., 2002; Hakimzadeh et al., 2006) and thin sugar juice (Shahidi and Razavi, 2006; Shahidi et al., 2006), using NF for recovery of salt from waste brine at a sugar decolorization plant (Salehi et al., 2011; Salehi and Razavi, 2012) and RO/NF clarification in the treatment of sugar beet press water (Shahidi et al., 2012), seems to find several applications there. On the other hand, some limitations exist for application of membrane processes in the sugar industry, since the volumes pumped are very high comparing to other food industry branches, the solutions exhibit high viscosity and high osmotic pressure.

7.1. *Sugar beet press water*

Press water derives from the pressing station of the extracted pulp after it passed through the extraction unit. Nowadays this stream is completely recycled to the sugar extraction unit. Press water typically contains 1–3% of total solids. The composition of solids is 60–80% of sugars, 20–40% of salts, colloids and suspended impurities. The amount of water is large, about 0.6 kg per kg of beet input, so during the processing of 10,000 ton of beet a day there recycles about 6000 ton/day of water, that brings back to the extraction process around 90 ton of sugar and 30 ton of impurities. Both components are undesirable, since their presence lowers the sugar extraction efficiency as well as juice purity and overall productivity (Bogliolo et al., 1996; Hinkova et al., 2002).

RO could provide permeate consisting almost of pure water to be used in the extraction unit and concentrate of all components, which can be sent directly to the low grade crystallization stage by-passing the high grade crystallization stage and can be promptly eliminated in molasses. Separation experiments were conducted on the two-stage RO system with pre-filtration step. Bogliolo et al. (1996) reported high retention RO membrane (OSMO 411T-MS10) in spiral wound module (4× 40″), NaCl retention 99%, at 3 MPa and 60 °C.

Nowadays when NF membranes are available, another flow-sheet, different from that proposed by Bogliolo et al. (1996), could be considered, where the first stage of RO is replaced by NF unit as it is indicated. It will result in two retentate schemes, the NF permeate being the input for RO stage. Then RO permeate will go back to the extraction stage. Recently Shahidi et al. (2012) used artificial neural networks (ANN) models for prediction of permeate flux and ionic compounds rejection during cross flow NF of sugar beet press

water based on the experimental data, which was obtained at different transmembrane pressures (TMP), temperatures and feed concentrations. The modeling results showed that the overall agreement between ANN predictions and experimental data was excellent for both permeate flux and rejections ($r = 0.998$ and $r = 0.974$, respectively).

7.2. Separation of green syrup colored matter

Sugar, as a final product, has to meet rigorous quality demands, so the general tendency is that the syrups from which sucrose directly crystallizes should have as low content of colored matter as possible, because they build up into crystals. In the process of sugar production, separation of non-sucrose compounds is carried in the process of liming and carbonatation and subsequent filtration. However, these operations involve high energy costs and result in the environmental pollution that cannot be neglected. Because of that the possibility of the application of new separation techniques, i.e. membrane techniques, has been intensively investigated. The interest in membrane filtration in the sugar industry has recently increased. Changing the existing technology needs large investments, so that is economically more effective to find such kind of membrane separation techniques that would alter some parts of the purification phase, or could be inserted into the existing technological process of sugar production (Decloux et al., 2000; Gyura et al., 2005).

Separation of colored matter from green syrup (intermediate product in the second stage of sucrose crystallization) was examined by UF and NF of a solution with 39.2% d.m. content by Gyura et al. (2005). NF was carried on a polymer membrane with MWCO of 0.5 kDa. Effects of NF at 30 and 50 °C, in the range of 5–30 bar were examined. Separation of colored matter on NF membranes was most efficient when the pressure was held on the level of about 30 bar and at a flow rate ranging from 300 to 400 l/h. Under these conditions, the permeate color decreased by 76%. The highest permeate flux (14.63 l m^{-2} h) corresponds to the range of TMP with the lower limit of 23 bar. Further, the enhancement of flow rate had a much weaker increasing effect on the flux, the highest values being observed above 360 l/h.

7.3. Recovery of regeneration liquid from decolouring ion-exchange resins

In the sugar industry, anion exchange resins are mainly used to remove high-molecular weight colorants such as melanins, melanoidines, caramels and polyphenols with molar mass in ranges from 0.5 to 20 kDa from sugar liquor. The colorants are at first adsorbed onto the resins and finally released from the exhausted resin into alkaline brine solution (100 g/l NaCl). One of the main drawbacks of an ion-exchange resin decolorization process is the production of wastes during the regeneration procedure. Wastewater from regeneration of decolorizing columns producing a stream characterized by high amounts of NaCl (50–100 g/1), organic matter (5 g/l as total carbon) and high chemical oxygen demand (COD) (13 g/l), which represents a serious pollution disposal problem and significant environmental costs (Cartier et al., 1997; Hinkova et al., 2002; Salehi et al., 2011). The production of highly colored salty effluents has found an answer with the use of membrane process. The retentate contains a concentrated fraction of COD which can more easily be disposed of and the permeate containing NaCl and small color bodies can be reused for regenerating the decolorizing resin. The potential of crossflow filtration to treat regeneration effluent was investigated by several workers. First tests with UF membranes result only in 45% reduction in organic matter and UF membranes were found not to be tight enough (Wilson and Percival, 1990). In contrast, NF membranes allow removal of small molecules without complete desalination and are thus becoming important tools for recovering small molecules or removing pollutants from effluent streams (Hong et al., 2006). NF process has been successfully employed to recycle anion exchange resin regeneration effluents in sugar industry (Wadley et al., 1995; Cartier et al., 1997; Durham et al., 2003; Hong et al., 2006; Salehi et al., 2011; Salehi and Razavi, 2012).

The NaCl retention of NF membranes is between 10 and 50%, while organic compounds retention has been reported by Wadley et al. in 1995 to be in the range of 80–97% on NF membranes (SelRO MPT-30 or MPT-31, Koch). It has been also resulted in 30% reduction in effluent volume and 60% reduction in salt consumption. In 1997, Cartier et al. demonstrated that with spiral wound membranes membrane (Desal 5.1 and Desal 5.2, and Filmtec NF4S) is possible to reach even 89% and 74% reduction in water and salt consumptions, respectively. Hong et al. (2006) proved that NaCl rejection decrease from 40% to 20% by increasing NaCl concentration from 0.01 M to 0.5 M.

Salehi et al. (2011) used AFC80 PA NF membranes (PCI membrane) to investigate the effect of feed concentration (40, 60, 80 and 100 g/l), TMP (1.0, 1.25, 1.5, 1.75 and 2.0 MPa) and temperature (30, 40 and 50 °C) on the recovery of usable brine obtained from waste brine in order to save on both salt and water consumption for ion-exchange regeneration process. A 77% reduction in salt consumption, a 90% reduction in water consumption and removal value of greater than 99% for colorants were reported. The rejection of NaCl was found to be 15–37%. The rejection of NaCl declined with increase in the feed concentration and temperature, whereas increasing the TMP led to an increase in NaCl rejection. The permeate flux was decreased by increasing the feed concentration and increased with increase in temperature and pressure.

The satisfactory qualitative performance and a higher reduction in water and salt consumption was reported by Salehi and Razavi (2012), (90% and 81%, respectively), using AFC40 PA NF membranes (PCI membrane). The average rejection of sodium chloride was found to be 9%. In addition, in this study ANNs were used to predict dynamically permeate flux and total hydraulic resistance (R_T) through the crossflow NF. The ANN was fed with 3 inputs: TMP, temperature and time. It was found that optimum ANN (3/9/2) configuration; gives the best fitting with the experimental data, which made it possible to predict flux and R_T with high correlation coefficients (0.96 and 0.98, respectively). Sensitivity analysis showed that among the input variables, pressure was the most sensitive factor for prediction of both flux and R_T.

This combination of resin and membrane processes (Fig. 2) is especially attractive for plant locations where the waste disposal is a critical issue. In the future, NF is expected to find application in recovery of water and salt from waste brine at a sugar decolorisation plant in sugar industry. In addition, the volume of toxic wastes discharged from sugar refinery columns can be lowered (Hinkova et al., 2002; Salehi et al., 2011).

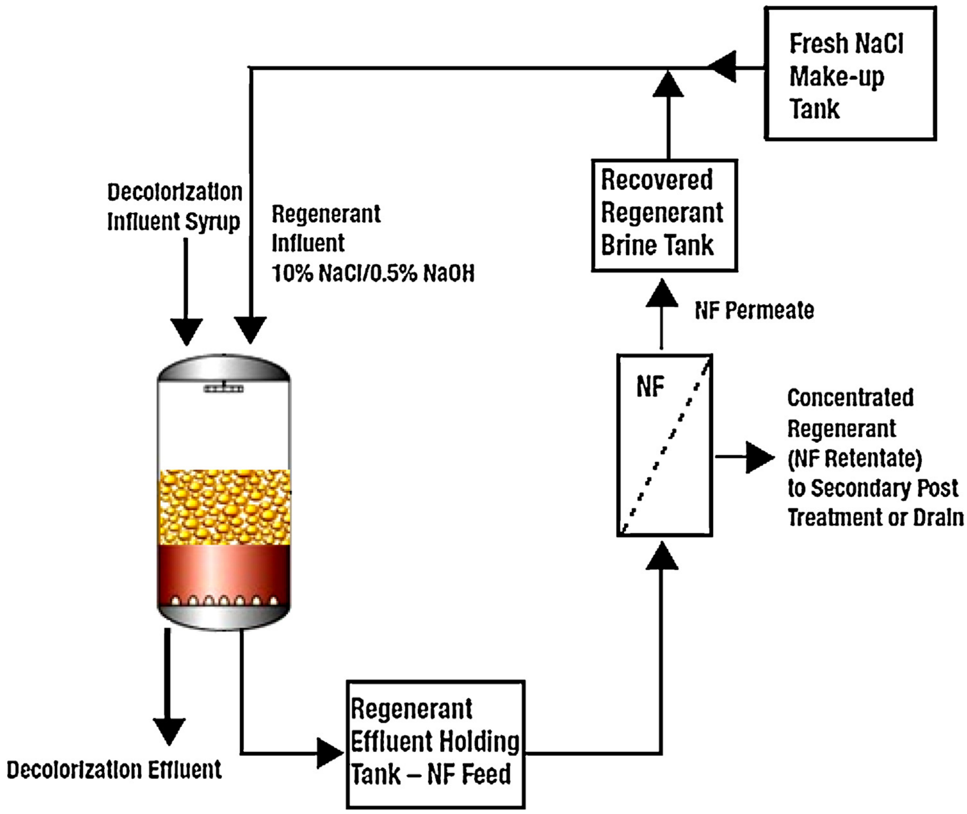

Fig. 2 – Schematic of NF process for purifying anion exchange resin regeneration effluent in the sugar industry.

7.4. *NF of xylo-oligosaccharide syrup*

Xylo-oligosaccharides (XOs) are functional oligo-saccharides with confirmed health benefits. In food industry, XOs were produced by chemical-enzymatic combination method. Main impurities in raw XOs syrup include monosaccharides (mainly arabinose and xylose), salts and organic acids, which can cause off-flavors and raise safety risks. NF is an effective method for the purification of XOs syrups by using membrane of proper MWCO between arabinose/xylose (150 Da) and xylobiose (282 Da) (Vandanjon et al., 2007; Mellal et al., 2008; Tsui and Cheryan, 2007; Hua et al., 2010). The application of membrane techniques in removing undesirable components and selecting XOs within given degree of polymerization (DP) rapidly increased in recent years (Pineloa et al., 2009; Hua et al., 2010). Also, its surface electrostatic properties allow inorganic salts to transmit (Chaabane et al., 2007).

Hua et al. (2010) results showed that the Nernst–Planck equation and film theory can be applied in predicting the separation performance of saccharides in XOs syrup and also help to understand the mass transport of saccharides in NF.

8. Vegetable oil processing

The conventional oil refining process is characterized by high energy requirements, losses of neutral oil, the need for large quantities of water and other chemical reagents, loss of nutrients and an elevated effluent production. Membrane based purification and separation technologies have been established as greater separation efficiency, cost effective (low energy consumption), reduction in number of processing steps, improved final product quality and environmentally friendly process for solid–liquid, solute–solvent and liquid–liquid separation applications (Subramanian et al., 2001; Baker, 2004).

With respect to the food industry, the focus of this technology is currently on the production of more adequate membranes for determined processes and products, or even to improve the quality of existing products (Cuperus and Nijhuis, 1993; Koike et al., 2002; Ribeiro et al., 2006; Ju et al., 2008; Sereewatthanawut et al., 2011). Recent research on the application of membrane technology to vegetable oils are, solvent recovery from the micella (mixture of extracted oil and solvent) (Koseoglu et al., 1990; Raman et al., 1996; Ebert and Cuperus, 1999; Wu and Lee, 1999; Geng et al., 2002; Kwiatkowski and Cheryan, 2005; Ribeiro et al., 2006), degumming (García et al., 2006; Kim et al., 2002; Moura et al., 2005; Koris and Marki, 2006; Marenchino et al., 2006; Pagliero et al., 2007; Subrahmanyam et al., 2006), deacidification (Snape and Nakajima, 1996; Kale et al., 1999; Zwijnenberg et al., 1999; Hafidi et al., 2005; Koike et al., 2002; Manjula and Subramanian, 2006), pigment removal (Koseoglu et al., 1990; Koseoglu and Engelgau, 1990; Subramanian et al., 1998; Reddy et al., 2001), wax removal using MF membrane (Mutoh et al., 1985), separation of emulsions (Kong and Li, 1999; Kocherginsky et al., 2003; Fontes et al., 2005; Del Colle et al., 2007; Hua et al., 2007; Ju et al., 2008), the synthesis and purification of structured lipids (Xu et al., 2000; Moura et al., 2007a, b), the separation of compounds present in trace amounts in the oil such as antioxidants (Sereewatthanawut et al., 2011). In general the most important characteristics of membranes are: permeate flow rate, heat, chemical and mechanical resistance, thickness, pore diameter, porosity and solvent permeability (Bhanushali, 2002; Coutinho et al., 2009). NF membrane technology shows that oil process with membrane, done under low temperatures, presents a promising alternative to conventional method toward the achievement of cost-effective

processes that are technically advanced and less toxic to environment.

8.1. Solvent recovery

The first vegetable oil processing step consists of the extraction of oil from oilseeds. The type of processing depends on the oil content of the oilseeds (Snape and Nakajima, 1996). Most of the oil extraction processes from oilseeds use hexane as solvent, what results in a miscella. The crude oil cannot be used without further processing, involves removing unwanted components and concentration of desired substances, and it need to refined to meet suitable properties for commercial applications (Koseoglu and Engelgau, 1990).

The solvent recovery from oil extraction (micella) process is the most efficient in terms of energy utilization, expanding ~20–25 kw h^{-1} per ton of soybean processed (Savasini et al., 1981) and about 1.7 kg of hexane vapor per ton of processed oilseeds is exhausted to the environment. On the other hand, with the NF technique, this amount can be reduced, at most, to the 5% level (Ebert and Cuperus, 1999). In the United States of America (USA) it has been estimated that about 2.1×10^{12} kJ per year could be economized by using a system based on membrane separation (Koseoglu et al., 1990). The application of membrane technology to separate the solvent from the micelles can be carried out soon after extracting the crude oil (Fig. 3). After filtering the micelle, the use of compatible NF would provide a hexane-rich current (permeate) that would be sent for recycling to the oil extractor, and an oil-rich current (retentate) that could be processed by distillation to recover the rest of the hexane.

Koseoglu and Engelgau (1990) reported the use of RO and UF membranes to recover solvent from the micelle. The membranes must resist the action of the solvents used and show high oil rejection. They proposed a combined process, the use of membranes with conventional distillation, for hexane recovery.

Raman et al. (1996) evaluated hexane-resistant NF membranes (Kiryat Weizmann, Israel) in the solvent recovery process from micelle consisting of 20% refined soybean oil. In the first stage, with a mean flow rate of 9 l m^{-2} h^{-1}, 24 °C and 2.76 MPa, they obtained a retentate with 45% oil. The mean permeate flux was 20 l m^{-2} h^{-1}, and the same pressure and temperature, obtaining approximately 99% oil separation with the combined system and reject more than 90% of free fatty acid.

Wu and Lee (1999) investigated the UF of soybean oil/hexane miscella using porous ceramic membrane. Raw soybean oil/hexane extract with 33 wt% of oil was used without pretreatment, using a membrane disk with a pore diameter of 0.02 μm and thickness of ~1 μm. The optimum separation conditions were found to be a TMP of 4 kg/cm^2 and 120 rpm agitation speed. The soybean oil concentration decreased from 33% in the feed to 27% in the permeate, that is, near 20% rejection.

Studies on membrane processing of crude vegetable oils showed excellent rejection of compounds, however, the permeate flux needed improvement for industrial adoption. Geng et al. (2002) investigated hexane separation from a crude soybean miscella (30%, v/v). Commercial ceramic membranes with different pore sizes (1, 3 and 5 kDa) were used, with a maximum pressure of 0.6 MPa. The membrane with a pore size of 1 kDa provided the best results with oil rejection of 70%, but with low permeate flow rates. Saravanan et al. (2006) studies on membrane processing of crude vegetable oils without dilution using nonporous membranes showed excellent rejection of phospholipids as well as color compounds, however, the permeate flux needed improvement for industrial adoption.

Commercial synthetic membranes are produced from two distinct classes of material: polymers consisting of organic material such as: cellulose acetate (CA), PA, PS and polyvinylidene difluoride (PVDF) amongst others; and inorganic materials such as: metals and ceramic materials (Cuperus and Nijhuis, 1993). Koseoglu et al. (1990) reported the use of NF and UF membranes in the separation of cotton seed oil (25%, w/w) from micelles containing hexane, ethanol and isopropanol as the solvents. NF membranes composed of PS, fluoride polymers, PA and CA with an average molar mass cut-off varying between 150 and 1000 Da were tested. Only the PA membranes (OSMO Sepa O, OSMO 192T-89, OSMO 192T-O, Osmonics) were stable to hexane. Iwama (1987) has found materials like aromatic PA, aromatic imide, PVDF and polytetra fluoroethylene (PTFE) can be found to be adequate.

Membrane instability can bring about unusually low or high fluxes due to crack-like openings on its surface and the shrinking or swelling of its structural matrix (Bhanushali, 2002).

Alicieo et al. (2002) evaluated the influence of temperature (50, 60 and 70 °C) and TMP on the crude soybean oil permeate flux through a ceramic tubular membrane (pore size 0.01 pm) and a polysulphone hollow fiber membrane (pore size 100 kDa). Pressures values are 3, 4.5 and 6 bar, for the ceramic membrane and 0.7 and 1.4 bar, for the polysulphone one. The permeate flux was lower for the ceramic membrane than the polysulphone one. For the ceramic tubular membrane, the best permeate flux was 4.16 kg/m^2 h, at 50 °C and 6 bar and for the PS hollow fiber membrane it was 11.58 kg/m^2 h, at 70 °C and 1.5 bar.

Separation of refined soybean oil/n-butane mixtures was studied by Tres et al. (2009) using different commercial UF and NF membranes, with cut-offs ranging from 1 to 5 kDa. The effects of the feed pressure (1–2.5 MPa) and the TMP difference (0.1–1 MPa) on oil flux and retention were investigated. Oil retention results ranged from 52.8 to 99.1% and n-butane flux up to 2730 g/m^2 h were obtained. Therefore, the NF membrane separation process has proven to be a promising alternative to the recovery of the hexane in vegetable oil extraction industries.

8.2. Deacidification

In the refining unit, the degummed oil is treated with a sodium hydroxide solution to react and precipitate the free fatty acids (FFAs) as soaps, and, at the same time, remove remaining traces of phospho-lipids. The refining units optimize the elimination of non-hydratable phospholipids and traces of metals by adding phosphoric acid prior to the treatment with soda, to avoid the formation of emulsions. The use of caustic solutions to remove undesirable unsaponifiable matter and FFA is effective, but may cause saponification of triacylglycerols (TAGs) and their removal in the soaps formed (Koseoglu and Engelgau, 1990; Koseoglu, 1991; Erickson, 1995). In theory, membrane technology solves the majority of these problems. The ideal process would use hydrophobic membranes, the appropriate NF membranes only allowing a partial separation of the FFA (Cheryan, 1998).

In another study, Snape and Nakajima (1996) used membranes to separate the lipids from hydrolyzed sunflower oil, and observed that the FFAs permeated preferentially through

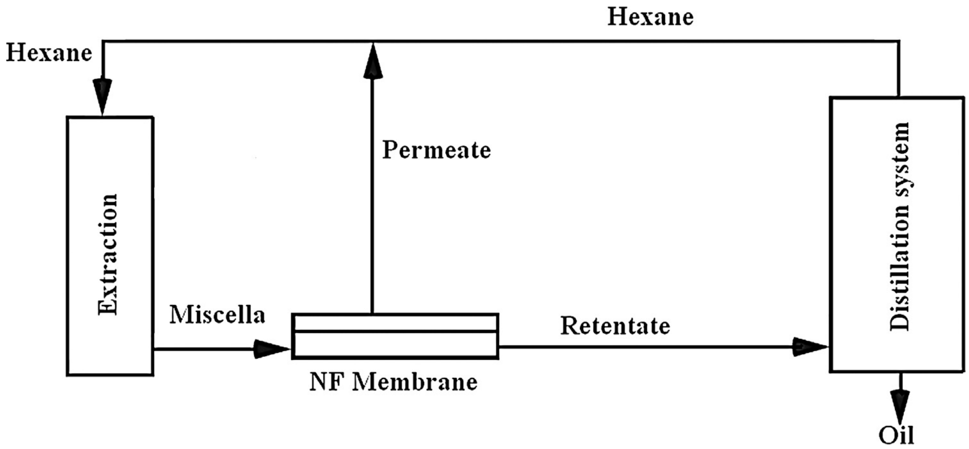

Fig. 3 – Schematic of the NF membrane process for solvent recovery after the oil extraction.

the membrane and concentrated in the permeate, whilst the TAGs were retained. Mono and diacylglycerols showed intermediate behavior, that is, they were equally distributed between the permeate and retentate.

Koike et al. (2002) used dense commercial RO membranes composed of cellulose acetate and of silicone polyimide, and managed to efficiently separate FFAs, monoacylglycerols, diacylglycerols and TAGs from high oleic sunflower oil hydrolyzed by lipases and diluted with organic solvents (ethanol and hexane).

In a combined process using refined soybean oil (TAGs source) and oleic fatty acid as feed in a filtration process through RO and NF membranes, combined with extraction by sub-critical pressurized fluid carbon dioxide, Lai et al. (2008) obtained the preferential permeation of oleic acid in relation to the TAGs. As from a model system containing 40% oleic acid and 60% TAGs (w/w), permeation through the RO membrane (BW 30) resulted in a permeate containing more than 80% (w/w) in oleic acid, whilst permeation through the NF membrane (NF 90, MWCO 200 Da) resulted in a permeate with approximately 50% (w/w) oleic acid. However this membrane showed a significantly higher permeate flow rate as compared to that obtained with the membrane BW 30.

Raman et al. (1996) selected six polymeric NF membranes (flat-sheet) for deacidification of methanol–oleic acid, methanol–mixed FFAs (oleic acid and linoleic acid) and methanol–crude rice bran oil (RBO)-FFA model solutions. They had high rejections (>90%) for oleic acid and >95% for a linolenic and oleic mixture. In addition, the rejection rate increased with the oleic acid concentration probably due to the formation of a fouling layer or a dynamic secondary membrane. In a similar study, Kale et al. (1999) used polymeric NF membranes for deacidification of methanol–extracted FFA obtained from crude RBO containing 1.7% phospholipids (PL), 16.5% FFA and 4% waxes. FFA in crude RBO was reduced to 0.33% (w/w, solvent free basis) after second stage solvent extraction and it was 0.06 and 20% (w/w, solvent free basis) in a third stage membrane permeate and retentate respectively. FFA recovered from the methanol extract was increased from 93% in one stage to 99% in three stages. On the basis of cost estimates and FFA recoveries analyses, they suggested a three stage plant as an alternative choice. A similar attempt was made to separate FFAs and TAG from the extracted phase that was obtained by alcoholic extraction of high FFA groundnut and RBOs (Krishna Kumar and Bhowmick, 1996). Membranes employed were CA (0.5 kDa, Amafilter GmbH, Germany), PS (1.0 kDa) and PA (0.5–0.6 kDa from Bhabha Atomic Research Centre, Bombay, India). Model mixtures of test samples were prepared by blending mixed FFAs and refined groundnut oil in different proportions and acid-degummed RBO with an FFA content of 34%. PA membranes were more suitable for FFA separation due to its slightly less hydrophilic nature and they were less prone to compaction with pressure than the CA & PS membranes used.

8.3. *Nutritional enrichment*

In an experiment simulating the refining of crude soybean and rapeseed oils without the addition of organic solvents, using polymeric membranes, Subramanian et al. (1998), reported that in a single step the filtration process managed to remove phospholipids, pigments and oxidation products. It also increased the tocopherol contents of the permeates by 12–26%, depending on the oil, as compared to the original tocopherol content in the crude oils, due to preferential permeation (negative rejection) of these compounds with respect to the TAGs.

According to Hafidi et al. (2005), the initial results showed the efficiency and potential of using membrane technology to neutralize olive oil. In a single processing step the oils were practically freed of FFAs and soaps. The process was carried out at room temperature, producing no polluting effluent. It was hoped to preserve the majority of the useful and heat sensitive components present in the virgin oil, and thus attend market needs and demands and avoid applying the complete refining process to these noble oils.

Crude rice bran is a natural source of γ-oryzanol, a nutritionally valuable phytochemical with antioxidant properties. In Sereewatthanawut et al. (2011) study, the refining and γ-oryzanol enrichment of RBO was investigated through solvent extraction optimization and NF processing. Several solvent resistant NF membranes (Starmem^{TM} and DuraMem^{TM} membrane series, Evonik, Ltd., UK) were screened and successfully applied in a two steps membrane cascade with fluxes between 39 and 53 $l\,m^{-2}\,h^{-1}$. Overall, the integrated process provided a RBO γ-oryzanol enrichment from 0.95 to 4.1 wt.% in oil, which corresponded to more than a two fold increase in the oil's antioxidant capacity. They results demonstrate the potential of organic solvent NF as a technology to enrich and refine oil

based products. The processes of solvent recovery, deacidification and the concentration of minority compounds in vegetable oils present great potential for exploration, since the studies carried out produced promising results, even with low permeate flow rates.

9. Conclusions

The increase in energy costs and the demands for products with greater nutritional value and of processing procedures less toxic to the environment are attractive factors for transferring membrane processing to food industries. NF membranes lie between RO and UF. It carries quite distinctive properties such as pore radius and surface charge density which influences the separation of various solutes.

NF membrane technology is increasingly used in treatment of water used in soft drinks manufacturing (Kewdale, Western Australia has Coca-Cola Amatil's (CCA) largest Australian NF plant). In desalination and drinking water applications, the NF has been used as a pretreatment process for RO and thermal desalination systems and removes the biofouling potential components, which results in increase in product recovery of RO process. There is also growing trend that utilization of dual NF in sea water applications results in reduction in energy and water cost. The review also carries the recent studies available in treatment of wastewater from food industries.

Recent advances and developments of the use of NF processes in the beverage industries are reviewed and discussed in this study. The potential advantages of membrane concentration techniques over conventional evaporation for concentrating fruit juice have been successfully demonstrated, including improved product quality, easily scaled up and reduce energy consumption.

The NF operation can successfully reduce the wastewater organic load and simultaneously contribute to the development of the cheese and curd cheese manufacture by-products. It can be expected that the expanding capabilities of membrane processes will continue to profoundly affect the dairy industry in the future.

In sugar industry, NF has been used in recovery of regeneration liquid from anion exchange resins in order to the reduction in salt and water consumption. In addition, the volume of toxic wastes discharged from sugar refinery columns can be lowered.

Utilization of solvent resistant NF membrane for processing organic solvents under different conditions in oil industries is also discussed. Recent research on the application of NF to vegetable oils has mainly targeted, solvent recovery, deacidification and the separation of compounds and it appears as an important alternative to conventional methods of processing vegetable oils.

Optimization of the permeate flow rates, rejection of compounds, reduction in membrane fouling and increases in the NF membrane lifetime appear as promising areas for future study and development.

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